United States Department of Agriculture
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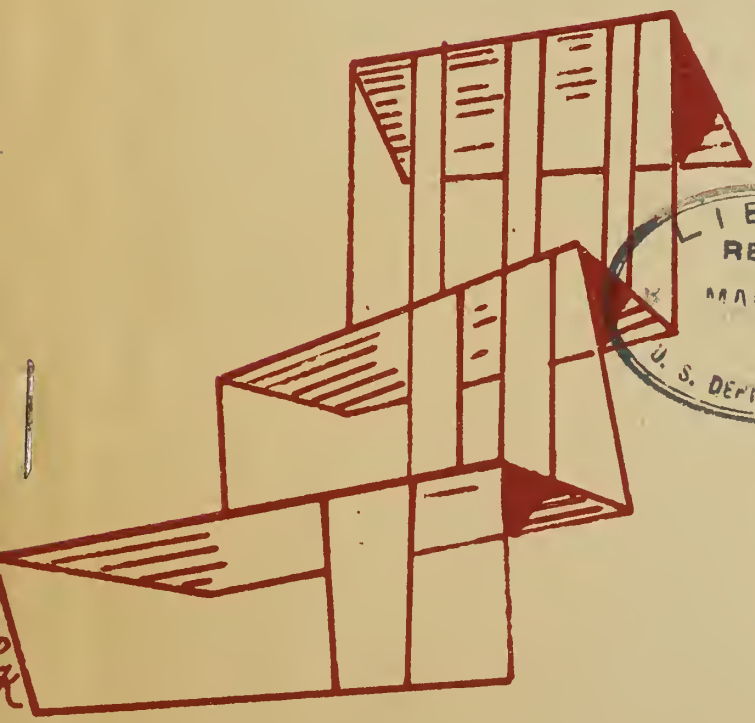
Bureau of Agricultural and Industrial Chemistry

advances in CROP UTILIZATION RESEARCH



at the

SOUTHERN REGIONAL RESEARCH LABORATORY
2100 Robert E. Lee Boulevard
New Orleans, Louisiana

1951

INTRODUCTION

This report indicates in a general way the fields of research receiving attention at the Southern Regional Research Laboratory 1/ in New Orleans, Louisiana, and at its field stations in North Carolina, Florida, Louisiana, and Texas, during the fiscal year 1951. No attempt is made to discuss each of the many research projects on which work has been done; but some of the more important accomplishments of the year, and commercial applications of research completed in previous years, are summarized.

These accomplishments may be classified into two categories: the solution of scientific and technological problems involved in the utilization of agricultural products and byproducts, and the acquisition by basic research of new scientific knowledge bearing upon the utility of agricultural materials. While the more striking advances in both categories are listed, they do not necessarily reflect the proportions of time and effort devoted to applied research and to fundamental research.

Research Results Applied Commercially

During 1951 the following accomplishments of the Southern Laboratory were adopted by various industries:

A new machine for opening and fluffing matted cotton from the bale, preparatory to cleaning and carding, is now in production by machinery manufacturers.

Tobacco shade cloth treated with lead chromate to retard damage to the cotton by sunlight has been made and sold commercially after its value to farmers was proved in cooperative service tests extending over three seasons.

Norconidendrin, a chemical made for the first time by the Southern Laboratory and shown to be effective as an antioxidant for vegetable and animal fats, has been produced on a limited scale by an industrial firm.

The first industrial installation has been made of a commercial self-cleaning filter modified in cooperation with an equipment manufacturer to adapt it for use in cleaning pine gum preparatory to steam distillation.

Information showing that the addition of citrus molasses to dried citrus pulp does not introduce a storage problem, under most conditions, is being utilized by feed manufacturers and dealers. A method developed for rapidly estimating the soluble-solids content of citrus feeds is used by feed mills for routine control work.

1/ The Southern Regional Research Laboratory and its field stations are part of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

Other Accomplishments

Significant advances in other research included:

Developed a chart that gives cucumber-pickle packers the exact proportion of sugar and vinegar required to put up sweet pickles so they will keep safely;

Confirmed on a commercial scale the role of enzymes in the spoilage of salt-stock cucumbers, and devised control methods;

Completed service tests to show that cottonseed-protein glues are satisfactory for bonding plywood intended primarily for interior use in dry climates;

Developed a starch product, composed mainly of abundant domestic starches, for use in making non-freezing electric dry cells to replace sweetpotato starch, which is no longer in commercial production;

Found that tung fruit dried to about a 12 percent moisture content does not absorb moisture under atmospheric conditions prevailing in the tung belt, hence fruit dried to this moisture will store safely and process efficiently;

Helped to develop ration candies in disk form suitable for packing in cans for the Armed Forces;

Developed and thoroughly tested a continuous steam still, having a throughput of 5 tons per hour, for producing turpentine and rosin from cleaned pine gum;

Produced from gum turpentine the new compound pinane hydroperoxide, which was found superior to the cumene hydroperoxide now used as a catalyst in making synthetic rubber.

Publications and Patents

Further information on these and other developments of the Southern Regional Research Laboratory is available in reprints of official publications, most of which were research papers published in scientific and technical journals. At six-month intervals the Laboratory issues a list of its published articles, with abstracts, for the information of interested persons. Single copies are available on request.

C O N T E N T S

Contents (continued)

I. COTTON LINT PRODUCTS

Textile Mills Using New Cotton Opener

A commercial-size machine has been developed at the Southern Regional Research Laboratory for opening and fluffing the matted lumps of cotton from the bale before cleaning and carding. In September 1950 details of the opening machine were released to the industry. As a result, the opener is now in commercial production and use. Three manufacturers of textile and cotton-ginning equipment are making it, and six additional licenses have been granted to machinery manufacturers and mills for use of the Government-owned patent. Four openers are in operation in the mills. One firm which bought two of the machines finds that their use has resulted in large savings. It reported: "We are sure it will pay for itself many times during the first year of operation." The savings will lower the manufacturing costs of textile products. It is estimated that savings up to $1 per bale on the lower grades of cottons processed are being obtained.

The new opener, with exceptionally high production (1 ton per hour) for its small size (requiring only 50 square feet of floor space), is a great aid in cleaning mechanically harvested cottons. The high speed of the opener does not significantly increase the formation of neps, and the grade and strength of yarns are unaffected. In addition to its value as an aid in cleaning, the machine reduces the amount of spinnable fiber removed with the trash by cleaning equipment.

Processing Methods for Reducing Neps and Improving Quality of Cotton Goods

Neps are minute, tangled masses of fibers formed in cotton during processing. They detract from the appearance of cotton fabrics and lower their quality where appearance is of first importance. Such defects are especially undesirable in fabrics that are to be dyed, for the neps dye to shades different from those of the body of the fabric.

Methods for substantially reducing the formation of neps during manufacture were discovered in research by the Textile School of the North Carolina State College of Agriculture and Engineering under a Research and Marketing Act contract with the U. S. Department of Agriculture. The work is being supervised by the Southern Regional Laboratory. Nep formation is influenced by fiber maturity, fineness, and length, as well as by excessive manipulation of the fibers during the opening, picking, and carding processes. It was found that neps can be reduced through careful selection of the raw stock and by making adjustments in the settings and speeds of the opening, picking, and carding equipment. The greatest improvement can be made at the card by applying methods which distribute the cotton in thinner and more even sheets on the cylinder and by closer settings between working parts of the card to prevent rolling and nepping of the fibers. Reducing the number of opening processes and reducing the beater speeds, or adjusting the rate of cotton feed to obtain milder treatment of the fibers, also will reduce nep formation.

Certain recommended measures have been adopted by a number of cotton mills, and substantial improvements in yarn quality have been reported.

Survey of the Luster Characteristics of American Cottons

American cottons can be selected effectively for the manufacture of end products in which maximum luster is desired as a result of work on improving the luster of cotton conducted by the Harris Research Laboratories in Washington, D. C., under a Research and Marketing Act contract with the U. S. Department of Agriculture. The work is being supervised by the Southern Laboratory.

For this research it was evident that the first step must be development of an accurate and dependable means of measuring or characterizing luster in textiles. Because the lustrous appearance of a yarn or fabric depends chiefly on the manner in which light is reflected from its surface, the method developed measures mechanically the light-reflecting properties of the cotton textile. It utilizes instruments and techniques usually employed in estimating gloss or reflectance of non-cotton materials, such as ceramics. The mechanically determined values of luster in textiles correlate well with the results of visual judgment in a wide range of materials.

A survey was made in which this method was applied to American cottons, embracing 103 varieties grown in 25 locations. The measurements showed that significant differences existed between varieties, but that each variety tended to hold similar rank from location to location in relation to other varieties grown at the same location. Correlation between fiber properties and yarn luster was found to be greatest for the X-ray angle and fiber strength, properties that relate to internal structure, and to be less marked for fiber length and fineness.

The new equipment, techniques, and knowledge developed in this study should aid further cotton research and breeding work and facilitate the selection of cottons for specific end uses.

Evaluation of Hopi-Acala 50 -- New High-Strength Cotton

A working plan for the evaluation of new and superior varieties of cotton has been developed through the coordinated efforts of such groups as the National Cotton Council and State and Federal agencies, including the Southern Regional Laboratory. The Southern Laboratory's part in the evaluation program includes determinations of special physical properties of fibers, mechanical processing characteristics, responses to chemical treatments including mercerizing and dyeing, and small-scale tests of the products in a limited number of selected end uses. A system of this kind can be used to speed the evaluation of essential properties and performance of the new varieties while they are still available in only small lots. The information gained will be of value to cotton breeders, and will also assist mills in evaluating some of the new cottons as they become available in larger quantities.

As a participant in this program, the Southern Laboratory investigated the properties of one of the new high-strength cottons developed by the Bureau of Plant Industry, Soils, and Agricultural Engineering, namely Hopi Acala 50, a variety that seems particularly suitable for growing in California. To determine whether the high strength had been obtained at the expense of other desirable properties, such as resistance to damage by flexing, tire cord manufactured from

the new cotton was evaluated. Wheel tests were run on tires made from Hopi Acala 50 cord and from a cord produced from a variety of comparable staple length, Stoneville 2B. The Hopi Acala cotton cord showed satisfactory flex life in tires as compared to that of the control. Evaluation of the mercerization properties of the new variety in yarn form showed that by mercerization it attained a higher degree of luster than Acala 4-42, the variety now grown in California, but there was no significant gain in strength by Hopi Acala over the gain by Acala 4-42.

The techniques developed in connection with the evaluation of Hopi Acala 50 will be applied in the evaluation of small lots of interspecies cottons, when experimental lots of these cottons are obtainable.

Importance of Cotton Waste Shown in Survey

A recent study of the production and utilization of cotton waste in the United States points the way for improving the efficiency of cotton-textile processing and utilizing more effectively the different types of waste produced.

This study, conducted jointly by the Southern Regional Laboratory and the National Cotton Council, showed that about 15 percent of all cotton consumed in the United States becomes cotton waste in the process of manufacture in textile mills. About 70 percent of the total cotton waste is fiber, the remainder consisting of motes, bits of leaf and stem, dust, and other foreign matter. In 1948 United States mills produced 642 million pounds of cotton waste having a value at the mill of $71,000,000.

The results of this study have provided a greater appreciation and knowledge of the size and nature of the waste industry, the factors influencing waste markets, methods for controlling waste, and opportunities for enhancing the value of cotton waste through research.

The large number of requests received for copies of this report -- about 1,000 in 8 months -- is evidence that this type of information is interesting and useful to a large part of the textile industry.

Reuse Value Shown for Cotton Fertilizer Bags

A recent development in the struggle of cotton-bag manufacturers to maintain cotton's competitive position has been the practice of supplying cotton flour and feed bags dyed and printed in attractive colors and patterns. Because their excellent reuse value was readily apparent, such bags immediately became popular, and this popularity has greatly helped in retaining an important outlet for cotton in the face of competition of lower-cost paper bags.

The success of this venture suggested to manufacturers the possibility of applying the same plan to the sale of cotton fertilizer bags. There was some fear, however, that cotton bags might be harmed by chemical action of the fertilizer with which they were filled, leading to loss of fabric strength or to fading of the colors used, or perhaps to both. After some debate on this point, the problem was referred by the Textile Bag Manufacturers Association to the Southern Regional Laboratory for investigation.

Representative samples of dyed and printed bag sheeting, as well as a quantity of commercial 5-10-5 fertilizer, were supplied by a member of the association. The laboratory filled several sets of small test bags with

fertilizer and stored them under different atmospheric conditions. One set was kept under conditions approximating those that would normally prevail in a warehouse in New Orleans; another was placed in a hot, dry atmosphere; and a third was stored in excessively warm, moist air. At regular intervals bags of each color and pattern were withdrawn for examination. At the end of a year there were no significant changes under any of the conditions employed. Experts in matching dyes could see no difference in color. Strength losses, by standard laboratory tests, were insignificant. Thus an indication that colored or printed cotton bags can be packed with fertilizer without appreciable damage to the color of the fabric was provided.

On the basis of results of the tests, the Textile Bag Manufacturers Association and the National Cotton Council have proceeded with their plans to encourage the use of dyed or printed cotton fertilizer bags. Their efforts, according to recent reports, have been attended with considerable success.

Weather-Resistant Tobacco Shade Cloth

Field trials have been completed by Florida tobacco growers of a weather-resistant lead-chromate treatment for cotton shade cloth applied experimentally by the Southern Regional Laboratory. The success of the trials has resulted in the commercial production of such treated fabric, which is now available in any desired quantity. This year one tobacco grower in northern Florida bought enough yardage of the treated cloth to cover a 10-acre field.

Increased durability of the treated cloth is responsible for the interest of tobacco growers. Samples first tested in 1948 have been used for three full seasons as top cover, and are being used a fourth season as wall cover. In contrast, untreated cloth is so rapidly degraded by the action of sunlight that it serves only one season as top cover and one as wall cover. The increasing price of cotton materials made shade cloth such an important item in the tobacco farmer's production costs that the demand arose for protective treatments to prolong its useful life. Commercial application of a lead-chromate treatment that effectively protects the cloth from sunlight is not a complicated operation, and the expense involved is small in comparison with the saving in replacement costs of untreated fabric.

Effects of Mechanical Processing on Fiber Properties

Knowledge of the relations between fiber properties and yarn and fabric qualities is important in research to improve the usefulness of cotton products. Recently interest has arisen in the significance of the changes that may take place in cotton fibers as they are processed from raw stock into yarn. Any damage to the fiber during processing would be carried over into the products and affect their utility.

Investigations have been completed in this field at the Southern Regional Laboratory and reports made of the effects of mechanical processing on the individual fiber strength, elongation at breaking point, and viscosity. The only significant change in processed cottons was decreased elasticity. In further experiments, ginned stock from seven commercial American cottons was processed

through the yarn stage. Again only the elongation and the modulus of elasticity, which depends upon elongation and is a measure of toughness, were affected; the elongation was consistently reduced, and the modulus increased. There was no evidence of damage to the fiber during spinning. This proof that spinning changes only the elongation properties makes it possible to use yarns rather than the unprocessed raw stock in eavluations of the various chemical treatments developed at the Southern Laboratory. Therefore the yarns prepared in the processing experiments have now been subjected to these chemical treatments for evaluation of their effects on physical properties of cotton yarns.

In an exploratory investigation under an RMA project, knowledge was obtained on the changes in the mechanical properties of cotton fibers that result from heating them in the presence of moisture. Fibers were heated for various periods in atmospheres of different humidities at 110°, 138°, and 162°C. Generally the loss of strength was greater as the moisture content of the atmosphere increased, but an atmosphere of 100 percent moisture -- live steam -- was considerably less destructive, probably because of the absence of oxygen. Data of this kind will be applicable to processes in which yarns or fabrics are subjected to changes in the surrounding atmosphere, as in dyeing and the application of size to warp yarns.

Advance Made in Testing Cotton Textiles

Although the cotton industry has for many years supplied the world with durable and satisfactory textiles in almost every essential civilian and military market, certain advances towards greater efficiency of operation and improved quality of products are needed. This need is heightened by the present state of national emergency. Cotton growers, merchants, processors, and research workers will be aided in their task of effecting the required improvements by having better testing methods and new information on the significance and the application of the tests. Improved testing methods and equipment could speed the work of the cotton breeder and research worker, lower the cost of processing control, provide new information on the relation between fiber properties and yarn and fabric performance, give improved products by indicating the best end uses for specific lots of cotton, and conserve supplies of cotton.

It is believed that the following advances in textile testing made by the Southern Laboratory the past year will have practical value for many laboratories and mills.

Improved X-ray technique for determining crystallite orientation

Fiber strength is closely with the orientation of the cellulose crystallites in the cotton fiber. For many years scientists have used this crystallite orientation, or the "X-ray angle," as an index of fiber strength. Such an index is particularly useful in selecting varieties of cotton for use as research standards and in guiding the development of new cotton varieties. The measurement of orientation, unlike the usual mechanical strength tests, may be carried out on a very small tuft of fibers, even those from a single seed. A more rapid method of determining the X-ray angle is provided by the development at the Southern Laboratory of a new rotating specimen mount for use with an X-ray spectrometer. The new procedure eliminates expensive photographic processes that are required

with other techniques, and, since it traces the X-ray curves automatically, dispenses with the usual time-consuming densitometer measurements.

Conditioning samples before testing

There has been considerable uncertainty as to whether cotton yarns and cords to be tested for breaking strength and elongation must be brought to equilibrium moisture content under the standard conditions (65 percent relative humidity at 70°F.) from a lower or a higher moisture content in order to obtain reliable results. This point has now been settled by experiments at the Southern Laboratory. Preconditioning of cotton yarns and cords before their exposure to the standard atmosphere for tests of breaking strength and elongation did not offer any practical advantage, and the expense of preconditioning all samples to approach moisture equilibrium from either the high side or the low side does not seem to be warranted.

Permeability of cotton fabrics

Although practical tests for measuring the permeability of cotton fabrics to air and water have long been available, much still remains to be learned about the underlying factors involved in achieving the desired fabric properties. It has been found that the pore-size distribution -- arrangement and size of void spaces in fabrics -- is one of the determining factors. This knowledge was gained by use of a special technique and an instrument devised for determining the amount of mercury that enters the dry fabric when immersed in mercury under pressures ranging from 1 to 600 pounds a square inch.

The technique was applied to learn the pore-size distribution in 18 cotton fabrics of selected closely woven constructions and to correlate the pore-size distribution with their known properties. Differences in the distribution were found to be related to differences in the maturity of fibers and tightness of weave and also to changes produced in the fabrics by scouring and finishing. Knowledge of this correlation between fabric properties and pore-size distribution helps to clarify problems encountered in research on developing airtight and watertight cotton fabrics. The method will be useful also for correlating the pore-size distribution with thermal conductivity and other properties that are dependent upon the void spaces in the fabric.

Mathematical expression for fiber roundness

One of several bases for predicting the behavior of cotton fibers in certain end uses is knowledge of their cross-sectional shape. All cotton fibers are twisted tubes, but the tubes can vary in shape from ribbonlike flatness to almost complete roundness. It is known that the roundness of fibers has an important influence on end-use characteristics of the fibers or final textile. Fiber shape, for example, is a good measure of maturity and affects luster, resilience, suitability for mercerization, breaking strength, and dyeing qualities. But one difficulty in establishing definite correlations has been the lack of a sound quantitative method for designating shape by means of a simple function that can be used as a numerical index.

Now a simple mathematical expression and an instrument have been devised in analytical and cotton-fiber research at the Southern Laboratory to evaluate the circularity of fiber cross sections and to measure the quantities defining the shape. The new technique, which has been used in connection with research to develop better water-resistant cotton fabrics, has general application in all investigations involving a correlation of shape and fiber properties. The instrument and the index in this way will be useful tools in gaining a broader understanding of the relations between individual fiber properties and the useful properties of yarns and fabrics. Several research agencies, including the National Bureau of Standards, have shown interest in using the new technique.

II. SOUTHERN OILSEEDS AND THEIR PRODUCTS

Research to Improve Hydraulic Pressing of Cottonseed

Hydraulic pressing, for many years the only process used to recover oil from cottonseed kernels, has lost ground to screw pressing, solvent extraction, and recently to the combination of forepressing and solvent extraction; however it is still used for more than half the cottonseed crushed each year. This method probably will continue to be important for a long time, especially for smaller mills that cannot justify the increased investment required to convert their operations to the newer techniques. For this reason improvements in the hydraulic-pressing method are being sought by the Tennessee Engineering Experiment Station under an RMA research contract with the Agricultural Research Administration, supervised by the Southern Regional Laboratory.

One of the major objections that processors have had to hydraulic pressing has been the amount of oil left in the press cake. Under the best operating conditions the press cake contains between 5 and 6 percent oil. The present investigations, conducted on a laboratory scale, have been directed specifically toward the solution of this problem.

If full advantage can be taken, on a practical scale, of the information provided in these experiments, there is a possibility that residual oil in hydraulic-press cake may be reduced about 1 percent below the average values. This would amount to an increase in cottonseed-oil production of about 10 pounds per ton of seed crushed. Not only would this add to the total dollar value of the products of hydraulic pressing, but it would also provide more oil that might be urgently needed under emergency conditions.

The investigation has included a study of different conditions for each step of the process, including variations in temperature and time of cooking, moisture content of the meats during cooking and pressing, the amount of pressure and rate at which pressure is applied, and the time and temperature of pressing.

Improvements in oil extraction on a laboratory scale were obtained by raising the temperature and lowering the rate and total amount of pressure. It was observed, however, that such changes did not bring about entirely independent effects, indicating that a combination of optimum conditions must be found to obtain the maximum benefits. Further work has been planned for this purpose.

Improved Process Found for Removing Color from Refined Cottonseed Oil

Some cottonseed oils, particularly those produced by screw pressing and refined by conventional methods, are not of acceptable color and quality for use in the manufacture of shortening. Such oils are often subjected to a second refining. Even after rerefining they still may not meet specifications for a shortening oil with respect to color.

Experiments at the Southern Regional Laboratory have demonstrated that the conventional rerefining process can be modified to produce acceptable products from these off-color oils. Essentially the modified process comprises the use of increased concentrations of caustic soda solution, and very high rates of agitation and shear in mixing the oil and alkali, with certain specified temperature limits. The changes from the conventional process, while simple, are radical, as the conditions found to be most effective for removing the color were previously believed to be deleterious to the oil and represented unacceptable refinery practice.

The optimum conditions under which the new process should be operated were established by systematically investigating the five variables affecting the process — concentration of the caustic soda solution, total amount of caustic soda used, speed of agitation, duration of agitation, and temperature.

Reduction in color of refined cottonseed oil was found to increase as the rate of agitation and shear of rerefining increased, up to an extremely high rate, beyond which further increases had little or no effect. About 0.2 percent of caustic soda (on weight of oil), used in the form of a 14- to 24-percent solution, agitation for 5 to 10 minutes, and a maximum temperature of 150° F. were found to produce maximum reduction in color. The color of the oils rerefined by the high-shear method usually was not more than half as dark as the color that would be obtained in the same oils by conventional rerefining. This proportional difference in color was also observed in the oils after bleaching. The high-shear rerefining method removed the acidic gossypol pigments of cottonseed oil more effectively than did conventional rerefining. In addition, the new method removed a high percentage of unidentified red pigments unaffected in conventional rerefining.

Aside from the fact that it requires some special mixing equipment and consumes more power than does conventional rerefining, the new process has no known disadvantage. In it conventional materials are employed, the oil requires no special treatment before or after rerefining, and rerefining losses are the same as with the conventional process.

Antioxidants Tested as Preservatives for Cottonseed Oil

During recent years a number of chemical compounds whose properties indicate they would be useful in stabilizing fats and oils against oxidative rancidification have become available. Most of them are phenols differing widely in chemical structure — some relatively simple, others complex. There has been considerable information on which to base the use of some in certain fields, but usually not enough to be a reliable guide to their effectiveness as antioxidants. The information on others, however, was meager, and data for evaluating the over-all usefulness of these new products were quite inadequate.

As part of its program of research to improve the utilization of oils from southern-grown oilseeds, the Southern Regional Laboratory in 1951 conducted an investigation aimed specifically at providing data for comparing the effectiveness of different antioxidants in stabilizing cottonseed oil against rancidity.

In previous research the Laboratory had demonstrated that cottonseed oil, when carefully processed and handled, has a high inherent stability toward oxidative rancidification because of certain natural substances (tocopherols) that are present. Hydrogenation, the process used in preparing oils for use in shortenings and margarines, further improves the natural keeping quality. Nevertheless, additional improvements are desirable if they can be attained.

The Laboratory tested 13 antioxidants for their ability to improve the keeping quality of a finished edible cottonseed oil and the same oil hydrogenated to shortening consistency. For comparison, the same antioxidants were added to prime steam lard, a product that is relatively low in natural antioxidants. As might be expected, they were much more effective in stabilizing the keeping quality of the lard than they were for either of the oils. Propyl gallate was found to be the best of the antioxidants tested for both hydrogenated and

unhydrogenated cottonseed oil. While less effective than propyl gallate, nordihydroguaiaretic acid and norconidendrin were found to be good antioxidants. Most of the other 10 antioxidants tested gave little or no increased protection to cottonseed oils, although they were effective with lard, even in small concentrations.

This study completed a thorough investigation in which much new and valuable information on the stability of cottonseed oil has been provided by the Southern Laboratory in recent years. Of particular significance are data explaining the mechanism by which fats and oils absorb oxygen and become rancid. Working first with the simple unsaturated fatty acid ester, methyl oleate, and later with cottonseed oil, the reaction of atmospheric oxygen with these substances was found to produce hydroperoxides, which on decomposition produced various saturated and unsaturated aldehydes that impart rancid odors and flavors to the fat or oil. This knowledge, obtained in work on cottonseed oil, applies also to peanut oil, and has been extremely valuable in developing the additional knowledge that is now available to improve the keeping quality of fats and oils in general.

Norconidendrin—New Oil Stabilizer—Made Commercially

Norconidendrin, a new antioxidant made from a compound that is recovered from the waste liquor produced during the pulping of western hemlock wood, recently has been made available on a limited scale by a commercial concern. This development is based on work carried out at the Southern Regional Laboratory in recent years and renewed in 1950 in cooperation with the manufacturer of the new product.

Evidence showing that norconidendrin has many potential uses has been obtained. For example, it is very effective in protecting fats and oils against rancidification. Small amounts added to cottonseed oil, peanut oil, and lard greatly increase the shelf life of these products. Other important possibilities include the stabilization of candies containing butter and of wax-paper coatings. Because norconidendrin appears to have many possibilities in the food and food-packaging fields, studies were started recently at the Western Regional Laboratory to learn whether or not the product can be used safely in edible products.

Other applications of norconidendrin have been made in the stabilization of unsaturated hydrocarbon distillates, lubricating oils, certain unsaturated insecticides, and oil-well drilling muds. Recent tests have shown that this product is highly effective also in retarding the deterioration of certain butadiene copolymers, as well as in the stabilization of vinyl-type monomers. Furthermore, it is reported to be effective in preventing the aging and discoloration of compounded rubber articles.

Many of the older commercial antioxidants are made from benzene and other chemicals that, because of the present emergency, are in short supply. Hence the use of norconidendrin, which is made from byproduct liquors of pulp mills, has the advantage of conserving critical chemicals.

The success of norconidendrin illustrates vividly the application of chemistry to enhance the natural properties of a product for greater usefulness. While searching for more effective antioxidants to prolong the shelf life of vegetable oils, Southern Laboratory chemists recognized a similarity between the chemical structures of compounds that had been used for this purpose previously and conidendrin, a substance known to be present in the wood of certain trees, including hemlock. Conidendrin itself was found to have only a slight antioxidant

effect when added to edible oils and fats. However, by altering the chemical structure of conidendrin, the chemists produced an effective new antioxidant, which they named norconidendrin.

Prior to this development, conidendrin had been a laboratory curiosity, although the possibilities for recovering it in large quantities as a byproduct of pulping western hemlock wood by the sulfite process on the west coast were known. Tests have shown that from 10 to 15 pounds of conidendrin can be recovered at moderate cost from the byproduct liquor of 1 ton of western hemlock pulp. Conversion of the conidendrin to norconidendrin is somewhat more costly, but at present the product sells for one-eighth the price of some of its competitors. Hence its use for certain purposes might effect a considerable saving in some of the industries depending upon antioxidants to insure the output of high-quality products.

Advances Made in Coordinated Research on Cottonseed Meal as Feed

Steps have been taken by the Southern Regional Laboratory to guide the planning of research to improve the nutritional value of cottonseed meal. By 1950 information had been obtained justifying the inauguration of a broad program of investigations under an RMA project to develop methods by which meals of higher nutritive value could be produced for more extensive use in the mixed-feed industry. Some of the results of this work are reported below.

Experimental meals suitable for hog and chick feeds

Investigations in cooperation with agricultural experiment stations and the cottonseed industry are pointing the way to unrestricted utilization of cottonseed meal as the protein supplement in hog and poultry rations, as well as in feeds for cattle. Preliminary results indicate that experimental cottonseed meals produced in commercial quantities by a modification of the screw-press method can be fed to hogs in concentrations as high as 43 percent of the total diet and to chicks in concentrations up to 70 percent of the diet without any harmful effect and with good support of growth. Heretofore recommendations for the use of cottonseed meal in these feeds called for concentrations of 10 percent or less for hogs and sometimes as little as 5 percent for chickens. These restrictions can be lifted as soon as meals equal in dietary value to the experimental meals are produced regularly by the industry.

Nutritive value related to chemical analysis and processing history

A definite relationship has been observed between processing conditions and the quality of cottonseed meal. Surprisingly, though, there is about as much variation among different meals produced by the same process as there is among meals produced by different processes. This was demonstrated in 1950 when samples of about 100 commercial cottonseed meals, produced by screw pressing, hydraulic pressing, and solvent extraction, were analyzed for free gossypol, nitrogen solubility, and other factors that might influence their nutritive value and were later fed to chicks and hogs. The extreme variability observed in the chemical analyses was confirmed by results of the feeding tests.

Even meals described as identical by present trading rules, which base the characterization largely on nitrogen content and color, were widely different in both chemical analysis and feeding results. Some of the meals were far

superior sources of protein than the usual characterizations indicated. As a result, nutrition investigators now insist upon information about the conditions under which meals are processed and their chemical analyses, in addition to the usual market description.

New approach to research on cottonseed meal as feed

With more and more investigators giving serious consideration to the effect of processing variables on the value of the product, an entirely new approach to research on the utilization of cottonseed meal is under way. Heretofore nutrition investigators have had the complete responsibility of procuring the meals, having them analyzed, and determining their nutritive value. Under such an arrangement it was difficult to give sufficient attention to the effect of processing variables.

Accordingly the Southern Regional Laboratory, in cooperation with the National Cottonseed Products Association, developed a new improved arrangement for nutrition research that provides for the cooperation of all parties involved from the production of the meal to its evaluation as feed. Under this arrangement, cottonseed meals are produced in commercial mills under conditions specified by the Southern Laboratory. The meals are analyzed and characterized at the Laboratory and then are furnished to nutrition investigators in State, Federal, and commercial laboratories. The arrangement makes it possible to correlate the results of feeding tests with the chemical analyses and processing history of the meals. Systematic changes can be made in processing conditions so as to study the effect to better advantage. In the last 3 years, 40 tons of experimentally produced cottonseed meals have been supplied for nutrition research under this program.

This coordinated program of research on cottonseed meals, with emphasis on processing history as related to nutrition, is paying off with information that promises to greatly increase the production of high-quality cottonseed meal. As the need for protein supplements for feeds and foods is unusually great in emergency periods, the acquisition of new knowledge on cottonseed meal is now particularly timely.

Usefulness of Cottonseed Meal for Plywood Glues Established

The Southern Regional Laboratory has completed a series of service tests on the use of glues made with protein obtained from cottonseed meal. The results confirm previous indications that cottonseed-meal glues, when they become available commercially, may be used in the plywood industry. Added to the information on hand, the service tests provide all the data needed by industry in judging the potential usefulness of the new cottonseed product.

The service tests were of two kinds -- "interior severe" and "exterior." Both were conducted under failure-accelerating conditions to speed development of the needed information. Plywood panels bonded with the glue were first subjected to the interior test, during which they were kept for 48 hours under warm, humid conditions (80°F. and 80 to 85 percent relative humidity) that were more severe than would ordinarily be encountered in indoor use. When this exposure was completed, the panels were allowed to come to moisture equilibrium in an atmosphere of 32 percent relative humidity at temperatures between 80° and 85°F. This was repeated for five cycles, and the reduction in bond strength noted. In the exterior test, samples were immersed in water at 75°F. for 48 hours and then dried. This cycle was repeated until the glue failed to hold the panels together.

The results showed that cottonseed-meal glues are entirely satisfactory for bonding plywood that is intended primarily for interior use in regions where the average relative humidity is low. Where the humidity is high, cottonseed-meal glues might not produce a sufficiently permanent bond for satisfactory use.

In its earlier experiments on the development of cottonseed-meal glues, the Southern Laboratory found that solvent-extracted meals produced with a minimum of heat gave stronger bonds than glues prepared from cottonseed meals made by other processes. Meals for experimental use were produced under ideal conditions in the Laboratory's pilot plant. When the usefulness of cottonseed-meal glue was definitely established, the studies were carried a step further to determine whether or not commercially produced solvent-extracted meal could be used. It was found to be satisfactory. As the number of plants producing solvent-extracted meals is increasing, there is no longer any question about the availability of raw material for the manufacture of cottonseed-meal glue on a commercial scale.

Control of Roasting Conditions Improves Quality of Peanut Butter

In continued research to determine the exact effect of processing conditions on the quality of peanut butter, the Southern Regional Laboratory, under an FMA project, has prepared butters from peanuts roasted under widely different conditions and has evaluated them both chemically and by tasting after storage at 80°F. for as long as 21 months.

These evaluations, together with the results of previous laboratory and pilot-plant studies, show definitely that careful control of roasting conditions is necessary for the production of peanut butter of optimum flavor and good keeping quality, and, furthermore, that the range in desirable roasting conditions is rather narrow.

New data also support previous observations that the oil contained in peanut butter is relatively stable to oxidative rancidity. Oils obtained from fresh peanut butters were found to have low peroxide values and long keeping times. Although some reduction in keeping times of oils extracted from peanut butters under storage was found to occur during the first 3 months of storage, the keeping times of these oils were still relatively long. No appreciable differences were found between the keeping times of oils from butters after storage for 3 months and those from butters after extended storage.

Taste-panel tests indicated a preference for medium-roasted butters stabilized with hydrogenated peanut oil for the prevention of oil separation, the average scores for such products being higher than those given to lighter-colored butters. In general, peanut butters stored at 80°F. and examined periodically by the panel were found to retain acceptable odor and flavor for as long as a year, after which the products developed objectionable flavors before any appreciable oxidative rancidity could be detected in the extracted oils. Butters made from the heavier-roasted peanuts tended to receive unsatisfactory flavor ratings at an earlier age than butters made from the lighter- and medium-roasted peanuts.

This information is of value to the peanut-butter industry because it makes possible the selection of conditions of processing that will produce butters having a high consumer rating and long shelf life. It is also of interest to the Quartermaster Corps because its application will assure the production of peanut foods of high quality.

Safe Moisture Content Found for Storing Tung Fruit Before Milling

Investigations at the United States Tung Oil Laboratory in Bogalusa, La., provided new information on the water-absorbing and water-holding properties of tung fruit that is useful for solving one of the major problems in utilizing the domestic tung crop. This problem is proper drying of the fruit and seeds to insure safe storage and efficient milling.

When the tung fruit falls from the tree it contains about 65 percent moisture and will heat, mold, and sprout if stored before it has dried to about 25-percent moisture. Even under favorable weather conditions, natural drying takes several weeks; sometimes in wet seasons the fruit never dries on the ground sufficiently to permit storage. Artificial drying of the whole fruit, on the other hand, is not practical.

Because the hulls contain more than half the total moisture in the whole fruit and have no value as a source of oil, their removal before drying or storage is desirable to decrease the heat required for drying and also reduce the space needed for storage. However, hulling followed by the usual storage procedure has some disadvantages that are discouraging to growers. It has been found, for example, that seeds broken in the hulling operation quickly spoil if stored in their normal moist condition. They develop free fatty acids rapidly and sometimes heat spontaneously. But research has also shown that tung fruit, seeds, and kernels tend to have their moisture contents balanced with that of the surrounding atmosphere. This means, of course, that knowledge of the equilibrium moisture contents of these products at different relative humidities can play an important part in their drying and storage; it was this knowledge that the Tung Oil Laboratory sought in its investigations.

It was found that tung fruit, seeds, and kernels in contact with air at 75 percent relative humidity (average for the tung-growing area) have equilibrium moisture contents of about 12.4, 8.2, and 6.1 percent, respectively. Consequently, if the products are dried to these moisture contents, they can be stored safely and processed efficiently. It is particularly significant that the hulls can be removed and the seeds dried to 8 percent moisture content with assurance that they will not spoil in storage.

In recent work the Laboratory has extended these investigations to determine safe storage conditions for tung hulls and press cake, which frequently spoil as a result of spontaneous heating. The results show that these products, too, can be stored safely if they are first dried to moisture contents in equilibrium with air at 75 percent relative humidity. These equilibrium moisture contents are 17.1 percent for tung hulls and 10.3 percent for press cake.

Fat Derivatives for Flexible Coatings Made from Monoglycerides

For a long time there has been a need in the food industry for an edible, highly flexible, nongreasy fat coating material to replace paraffin and other nonfat coatings now used. In general, flexibility and lack of greasiness have been mutually exclusive properties of fats. A nongreasy fat is usually hard and brittle; a flexible fat is usually soft and oily.

Recently, however, the Southern Regional Laboratory, in work under an RMA project, developed flexible and nongreasy fat derivatives that can be prepared in a fairly simple manner. Monostearin, a monoglyceride prepared from glycerin and completely hydrogenated cottonseed oil or similar fat, is allowed to react with acetic anhydride, an inexpensive commercial chemical that unites with water to form acetic acid. The monostearin and acetic anhydride are heated together for about 1 hour at a temperature of about 230°F. The product of this reaction, acetostearin, needs only to be washed with water and in some cases deodorized prior to use.

Various acetostearins having melting points ranging from 80° to 140°F. can be produced. The melting point of any particular acetostearin depends on the grade of monostearin used and the degree to which it is permitted to react with the acetic anhydride. Generally, such products do not have an exact melting temperature, but melt within a temperature interval of 2° to 5° Fahrenheit.

Below their melting points and down to room temperature most acetostearin products are nongreasy and flexible. Below room temperature, most of the experimentally prepared acetostearins could be stretched more than 800 percent without breaking. Even the least flexible acetostearin product stretched six times as much as did paraffin wax (melting point, 125°F.). At freezing temperatures the acetostearin products are still flexible.

Although no known facts indicate that these flexible fats are not edible and digestible, they are being carefully evaluated in this respect by several laboratories before being recommended for use in foods. Their possible use as coatings for cheese, dressed meats and poultry, ice-cream bars, and other food products is being explored, along with other interesting possibilities in nonfood fields.

Efforts Made to Improve Storage and Processing of Rice Bran for Oil

Interest in the production of rice-bran oil as a byproduct of the rice-milling industry has grown rapidly in the past few years, largely as a result of technical information made available by the Southern Regional Laboratory. The oil can be extracted readily and economically, and, if extracted from fresh bran, its quality is excellent. However, several problems remain to be solved before all the available bran can be utilized as a source of oil. Their solution was the objective of further work on rice bran under an RMA project.

Safer storage procedures

One of the problems stems from the fact that rice bran does not keep well between the time it is removed from the grain and the time it is processed for oil. This has resulted in a serious storage problem because facilities are inadequate for immediate extraction of all the bran available.

When rice bran is stored free fatty acids form rapidly, so the oil deteriorates in a very short time. To solve this problem, it is necessary first to understand the cause. For this reason the Southern Laboratory has been conducting a thorough investigation of the factors that contribute to the formation of free fatty acids in rice bran during storage. Evidence has been obtained that the rate at which the free fatty acids form depends on the moisture content of the bran. However, the biological factors responsible for the deterioration of rice-bran oil occur in the bran itself or in the micro-organisms found in the bran. During the past

year, therefore, an investigation was made of the micro-organisms in bran as related to moisture content and to the formation of free fatty acids.

Although the investigation is not complete, because information is not yet available on the effect of the enzyme systems of the bran itself, information has been obtained that should be of practical value to the rice-bran-oil industry in developing means of storing the bran without deterioration.

For example, experiments on sterile bran inoculated with selected strains of mold and bacteria, demonstrated that these agents can cause the development of free fatty acids in the bran oil and, therefore, must be controlled in order to completely prevent deterioration during storage. It was observed further that moisture hastens the growth of both molds and bacteria. Molds grow fastest when the relative humidity is about 80 percent; bacteria, at 100 percent.

The obvious conclusion is that rice bran should be stored in as dry a condition as possible. While the moderate heating normally used to dry rice bran was found to destroy very few of the micro-organisms present, and apparently did not affect the enzyme systems at all, such heating did remove the water that permitted hydrolytic reactions involved in the formation of free fatty acids. Thus, if bran dried in this manner could remain dry, deterioration would be prevented. However, it was observed that whenever the dried bran was exposed to the high humidities prevailing in the southern rice-growing areas it absorbed moisture, permitting the natural systems of micro-organisms in the bran and oil to promote deterioration.

Heating the bran with steam under pressure killed all micro-organisms and removed the natural tendency for the bran to deteriorate. Regardless of the moisture content, free fatty acids will not form in bran that has been thus sterilized and maintained in a sterile condition. Sterilization followed by dehydration, therefore, would seem to be a method for complete control of deterioration during the period when rice bran must be stored prior to solvent extraction. Even if the bran absorbs some moisture during this period, the onset of deterioration will be slow.

Better refining techniques

In another line of research the Southern Laboratory obtained information to improve techniques for refining rice-bran oil. In the past there have been difficulties, especially when the oils were high in free fatty acids, as they often were when the bran had been stored for considerable time prior to extraction. An experimental procedure that involves steam stripping of the oils seems promising as a solution to this problem. The crude rice-bran oil is degummed and dewaxed in the usual manner; then it is treated with steam before the usual refining step, in which an alkali is used.

In pilot-plant experiments steam stripping reduced the free fatty acid content of rice-bran oil from about 9 percent to less than 1 percent and lowered refining losses enough to yield 8.5 percent more oil. Additional studies are under way to determine the best conditions for practical application of this procedure.

Basic information for processors

To assist the industry in considering the economic aspects of rice-bran-oil production, the Southern Laboratory has made a study to obtain more definite information on the amount of oil that might be expected from different lots of bran. Samples of bran from eight commercial varieties of rice grown in Texas, Louisiana, and Arkansas have been analyzed to determine the influence of variety and environment on the oil content. Purified bran (comprising the true pericarp and germ fractions) contained from 18 to 25 percent oil, depending more on the variety of rice than on the locality where grown. The amount of oil in commercial rice bran is lower because the milling operation removes some of the inner starchy part of the grain (endosperm) along with the bran. Variations in milling, as well as in physical condition of the grain, affect the amount of oil.

III. STARCH PRODUCTS

Replacement Starch Developed for Nonfreezing Electric Dry Cells

In the manufacture of voltaic cells of the dry type found in some radio, telephone, and other communication equipment, in flashlights, and in weather-recording instruments, starch having certain properties is an important raw material. It is widely used in preparing a special coating that separates the outside zinc can (negative electrode) from the pasty mixture of carbon, manganese dioxide, and other chemicals that fills the can and surrounds the carbon (positive) electrode in the center.

When dry-cell batteries are manufactured for use at very low temperatures, a special process is necessary to prevent "freezing up" and failure to operate. In this process a starch that makes a paste of certain special properties must be used. High-quality sweetpotato starch made by a process developed by this Bureau had these properties. During World War II, and until recently in fact, this was the only product found satisfactory in the manufacture of dry cells of sufficiently high quality to meet the rigid requirements of the Signal Corps for use in equipment of the armed services in subzero weather.

The commercial production of sweetpotato starch ceased in 1947, because prices did not favor the use of this vegetable as an industrial raw material. The problem of a continuing source of satisfactory paste for electric dry cells for military use at very low temperatures became acute. The Southern Regional Laboratory was asked to study the situation and make suggestions based on its experience in the development of high-quality sweetpotato starch.

By applying available knowledge of the physical and chemical properties of sweetpotato starch, a formula was developed for a new product composed essentially of abundant domestic starches which was believed to have the properties needed for making nonfreezing dry cells. This mixture has now proved satisfactory for commercial production of high-quality dry cells that are acceptable to the Signal Corps for use in extremely cold weather.

Thus the Laboratory's research, which led to a successful method of producing sweetpotato starch under favorable economic conditions, has also provided a replacement for sweetpotato starch for use in an essential industrial and defense product when sweetpotato starch is not available commercially.

IV. SUGARCANE AND ITS PRODUCTS

New Method Developed for Using Two Sugarcane Byproducts

The cane-sugar industry needs new sources of income to maintain profitable operation in the face of increasing costs. Sugar-milling operations are so efficient that further process improvements offer little opportunity for significant cost cuts. However, improvement seems possible in the utilization of the industry's primary byproducts — blackstrap molasses and bagasse (cane refuse after the juice is extracted for sugar making). The Northern Regional Laboratory has proposed a method for such improvement.

Most mills utilize the byproduct bagasse in only one way — for fuel. However, advances in sugar-house heat economy leave a large surplus of bagasse that is not needed to generate steam. Usually this excess is burned simply to dispose of it. Several sugar mills in the United States bale bagasse and sell it to the insulating-board industry at a small profit. A few mills convert the byproduct into such commodities as plant mulch and chicken litter. In all, however, only a negligible proportion of the total bagasse in the world is used profitably.

Blackstrap molasses, the second cane-sugar byproduct, has not found a stable market price for years. Although its use for livestock feeding is increasing, its price has been based on the alcohol market. The sugar industry wants to tie blackstrap molasses in with the feed market because its highest value is for feed.

The most satisfactory solution of byproduct problems is for the industry producing the byproducts to take upon itself the task of converting them into high-grade merchandise with reasonably stable and larger markets. All the successful industrial uses of bagasse and blackstrap have been developed independently of the sugar industry. Making the most of the combination of blackstrap and bagasse is obviously the task of the sugar mill. The results of cost studies and small pilot-plant work at the Northern Laboratory point to the joint conversion of blackstrap and bagasse into new, higher-grade products as the best solution of the sugar mill's byproducts problems.

Basis of the conversion process proposed by the Laboratory is the method it has developed for separating pith and fiber directly as the bagasse comes from the cane mill. Both pith and fiber are then dried. The quantity of pith is sufficient to absorb all the molasses, forming a nonsticky mixture that can be used as a component of animal feeds. The dried and baled pith-free fiber is available for sale to paper mills.

Although the method has been tested only in the Laboratory's pilot plant, the preliminary data have been presented promptly to the industry. Engineering design and data on operation are being obtained for large pilot-plant installations at sugar mills.

Estimates of costs and profits of the process are based on assumptions which must be further verified. Indications are, however, that the operation may give the sugar mill an additional profit of near $1 per ton of cane processed.

When the pith-free bagasse, obtained by the new method, is pulped it yields a superior material for making paper — an especially significant discovery in view of the serious pulp shortage developing in the United States. The pith-molasses mixture appears to be a usable feed for cattle, particularly at a time when cattle production is increasing in the South.

Processing of New Sugarcanes Studied

New high-yielding, disease-resistant varieties of sugarcane, and varieties with improved adaptability to mechanical harvesting, are being planted by more and more growers. This change to new varieties makes obsolete much of the existing information on the processing and milling characteristics of commercial sugarcanes. From earlier experience it can be assumed that existing information cannot be used as a guide to the behavior of the newer canes as to efficiency of juice extraction, effectiveness of clarification, and yield of sugar. This has been demonstrated repeatedly in commercial operations, but such operations are on too large a scale to determine the cause. Commercial mills necessarily handle mixtures of canes of widely different qualities, so it is not practical to study the juice from any particular lot separately.

For this reason the Bureau, under an RMA project, has made arrangements to conduct complete pilot-plant investigations in order to get detailed information on the effects of special properties of the new canes and their juices upon the recovery of sugar. These arrangements include an RMA contract for milling tests by Louisiana State University at Baton Rouge, followed by studies of the subsequent processing steps by the Southern Regional Research Laboratory. These studies are conducted jointly in a special pilot plant provided by the University.

Experiments can be carried out on individual lots of cane weighing as little as 2 tons; yet the processing is continuous, and operating conditions are automatically controlled to simulate those of full-scale sugar manufacture. In this way it is possible to study the processing behavior of juice from a particular lot of cane, and to correlate results of the experiment with the composition of the cane and juice used. The relatively small amount of material required also makes it possible to conduct tests on some of the more promising unreleased varieties of cane to obtain advance information on any peculiarities of composition that could affect ease of processing and recovery of sugar.

In the program conducted in the 1950 grinding season, two or more experiments were made on each of eight varieties of cane supplied by the American Sugar Cane League. These varieties included the newer commercial varieties in Louisiana and two that have not yet been released for commercial planting. The behavior of these canes in milling, including the power required for grinding them and the efficiency of sugar extraction, were determined in the milling tests conducted by staff members of Louisiana State University.

Juice obtained from each variety was collected and kept at a low temperature to permit continued processing at a rate of about 50 gallons per hour. In the small-scale liming and clarification equipment, the experiments conducted by the Southern Laboratory revealed differences in rate of clarification, volume of impurities precipitated, and clarity of the juice. In some of the experiments observations were made on the ease of filtering the precipitate obtained in

clarification, on the ease and completeness of crystallization of sugar, and on the quality of raw sugar produced. These data were correlated with analyses of the original juice and of the sirups and molasses resulting from its processing. The information was published promptly to help the operators of Louisiana sugar mills.

Thus far attention has been given primarily to a comparison of the newer varieties of cane. A broader objective of the investigations is the study of the exact manner in which milling and processing generally are affected by the composition of sugarcane and of the juice produced from it under various milling conditions. With increasing knowledge on the minor constituents of cane and juice and the development of new analytical techniques, it will be possible to determine precisely which constituents or classes of substances are responsible for difficulties in the clarification of juice and crystallization of sugar. This will serve as a basis for developing methods to overcome the difficulties and to obtain more effective clarification, greater recovery of sugar, and better quality.

Knowledge of Composition of Sugarcane and Products Expanded

While the mineral (ash) constituents of sugarcane, cane juice, molasses, and sugars have been extensively studied, knowledge of the organic nonsugar constituents has remained limited. Yet such knowledge is greatly needed as the basis for a better understanding of the chemistry of sugarcane processing, which should lead to improvements in the operations involved. During the past year the Bureau began investigations under an RMA project to fill this gap in the available information by the application of modern research methods.

The results of the new approach to the problem already have more than doubled the number of simple organic nonsugar substances known to be present in sugarcane juice, and are expected before long to provide the data necessary for characterizing the more complex substances.

The studies have been made partly under an RMA research contract by Ohio State University and partly by the Southern Regional Laboratory.

Scientists at the university isolated several complex polymeric substances containing nitrogen from molasses made from domestic sugarcane juice in which tryptophan and lysine, the simpler amino acids, were known to be present. Using ion-exchange and chromatographic methods, they were able to detect at least small amounts of glutamic acid, glutamine, serine, glycine, alamine, and gamma-aminobutyric acid in this same juice. Isolation and complete identification of these hitherto undetermined constituents of cane juice are now in progress. New information of this kind on the nitrogen-containing amino acids is important, because these constituents, during processing, are believed to form complex substances that increase the amount of molasses and decrease the amount of sugar produced.

At the Southern Laboratory studies were directed toward the isolation and identification of the organic acid constituents of cane juice that do not contain nitrogen. This work was done in connection with studies of aconitic acid, which is the principal organic acid constituent of sugarcane and which, by means of a process developed by the Bureau, is now being recovered commercially as a by-product of sugarcane processing. In addition to aconitic acid, only malic, oxalic, and possibly citric acid had been previously identified in the nonvolatile matter

isolated from sugarcane juice. By chromatographic fractionation on a silica gel column the Laboratory has now effected the separation of not less than five other organic acids of this class. Two of them have been purified by crystallization and definitely identified as succinic and fumaric acids.

This investigation has disclosed certain errors in conclusions reached by the application of older and more cumbersome analytical methods. Scientists formerly thought most of the aconitic acid in molasses was lost in the course of fermentation for the production of alcohol, and that succinic acid found in the still residues was a product of the fermentation. Definite information to the contrary is now available. By means of its own accurate method of determining aconitic acid, and by the application of new chromatographic methods, the Southern Laboratory has shown that approximately 85 percent of the aconitic acid in molasses can be found in the still residues after fermentation to alcohol and that succinic and fumaric acids, originally in the molasses, also accumulate in the fermentation residues.

Improved Candies Made for Military Rations

This Bureau, in cooperation with the National Confectioners' Association, has worked closely with the Chicago Food Laboratories of the Quartermaster Food and Container Institute on the development of improved candies for use in rations for the Armed Forces.

There is need for confections that combine high concentrations of various nutrient substances with greatly improved storage life and acceptability to the soldier in the field. A wide variety of candies, as nearly as possible like the candy bars and other confections to which the troops were accustomed as civilians, is wanted. The problem is to prepare such popularly acceptable candies to meet the packaging and storage requirements of the Quartermaster Corps. The candy must keep safely for 6 months at 100°F. or 2 years at or below 70°F. Much of the candy for the Armed Forces will be packed in cans, and therefore must be in disk form. Moreover, the candy should be of kinds suitable for starch molding in order to take advantage of the large capacity of the confectionery industry for producing molded candies.

Among the improved candies developed thus far at the Southern Regional Laboratory for evaluation by the Quartermaster Corps are disks of fudge, caramel, and caramel-nougat combination.

Sorbitol, a new ingredient that is obtained by the hydrogenation of corn sugar, has been added to the fudge to keep it from drying out, which is the principal cause of deterioration in this candy. A fudge of excellent texture, which should retain its smoothness much longer than standard fudge preparations, is produced by the use of 10 percent sorbitol, with proportional reduction in the amounts of sugar and corn sirup.

The new caramel, which is of such outstanding quality that the Quartermaster Corps considers it particularly desirable for military rations, resulted from experiments with the solids from sweet whey, recovered in dry powdered form as a byproduct of cheese manufacture. This candy is prepared in such a way that it can be cast in starch molds in the disk form required for packaging in cans. It can thus be produced rapidly and with little labor.

The combination candy in disk form, which is very similar in other respects to a high-quality candy bar, was made by using a layer of nougat with some of the caramel to which dried sweet whey was added.

The coating on all these candies is a regular commercial summer-type coating chocolate that withstands heating at 110°F. for 2 years and does not melt readily when the candy is eaten. Further research is directed toward the development of a chocolate coating of better eating quality that will also meet the Quartermaster Corps' severe temperature requirement.

Storage tests are in progress to determine whether the high initial quality and acceptability of the candies so far developed will be retained when they are subjected to the tests applied to ration items.

V. CUCUMBERS AND PICKLES

Sweet-Pickle Formulas Standardized to Prevent Losses

A chart that gives cucumber-pickle manufacturers the exact proportions of sugar and vinegar required to put up sweet pickles so they will keep safely was worked out in 1950 by the Food Fermentation Laboratory in Raleigh, N. C., in cooperation with the North Carolina Agricultural Experiment Station.

Information of this kind has been greatly needed. The procedures formerly used were based, for the most part, on each packer's experience. They were far from uniform or reliable, as evidenced by frequent cases of gaseous spoilage--the result of too little sugar or vinegar, or both.

One of the limitations in establishing uniformly safe preservation practices has been the lack of a suitable test organism having a wide range of tolerance to sugar, acid, and combinations of these ingredients. The Food Fermentation Laboratory obtained a number of cultures of a yeast (*Zygosaccharomyces* species) from typical cases of sweet-pickle spoilage in the industry. When this yeast exhibited rather remarkable resistance to sugar solutions and vinegar, individually in various concentrations, and to mixtures of the two, the Laboratory subjected it to a thorough investigation under carefully controlled conditions. This investigation led to the development of what is believed to be the first preservation-prediction chart available to the pickle industry.

The chart has been tested in a commercial plant in the making of about 40 experimental batches of sweet pickles over a 2-year period. Pickles made with the proportions of vinegar and sugar recommended on the chart did not ferment; those made with less of the preservatives underwent gaseous fermentation and spoilage. Laboratory examination of each spoiled lot showed yeast to be the causative agent. No doubt this chart, now being prepared for publication, will find wide-spread use in the pickle industry for standardizing sweet-pickle formulas to reduce spoilage and save sugar.

Cucumber Softening by Enzymes Controlled in Pickle Plant

Previous research reports suggested that the cucumber itself may play an important part in the undesirable softening that sometimes occurs in this vegetable during the period of storage as salt stock which precedes the manufacture of brined pickles. This theory was based on fundamental studies in which the nature and occurrence of a pectin-splitting enzyme in the cucumbers themselves and in various parts of the plant were thoroughly investigated by the Food Fermentation Laboratory in Raleigh, N. C., in cooperation with the North Carolina Agricultural Experiment Station.

The fundamental data have now been confirmed in a commercial-scale experiment conducted in cooperation with a pickle manufacturer. Some 15,000 bushels of Palmetto variety cucumbers (produce or slicer type) from the fall crop of 1950 were brined during cold weather under conditions that precluded fermentation. When these cucumbers showed signs of softening within a few days after the stock was covered with a 10-percent salt brine, immediate tests were started to discover the reason.

The Food Fermentation Laboratory obtained samples from 15 vats of the brined cucumbers and analyzed them carefully along with samples of fresh Palmetto cucumbers obtained from two growing areas (Florida and South Carolina). A thorough study of all these samples, and a comparison of the results, demonstrated that the softening was caused by a pectin-splitting enzyme that occurs naturally in the cucumber. This particular enzyme was found in samples of brine taken from vats in which the softening cucumbers were stored and also in various sections of the brined cucumbers. In addition, it was found in different parts of the fresh, unbrined cucumbers of the same variety.

The immediate result of the study, of course, was the saving of the 15,000 bushels of cucumbers affected, but the findings are even more significant from a long-range viewpoint. After identifying the responsible enzyme, the Food Fermentation Laboratory found that it was sensitive to acid and could be destroyed by treatment with either lactic or acetic acid, or inactivated by heating the brined material. Prompt application of these treatments by the manager of the cooperating plant checked the softening before serious losses had occurred. This verification on a commercial scale of the data previously developed in fundamental research on the role of enzymes in the spoilage of cucumbers is another step forward in efforts to improve the processing of food products. The control methods applied in this particular study will be valuable to the entire pickle industry as situations involving losses from the softening of salt-stock cucumbers arise.

VI. CITRUS PRODUCTS

Effect of Storage Temperatures on Frozen Citrus Concentrates Studied

Studies that will aid processors and handlers in the storage of frozen citrus concentrates have been undertaken in cooperative work under an RMA project by the Citrus Products Station at Winter Haven, Fla., and a commercial processor of citrus fruits.

The presence of some well-distributed pulp and cloud in citrus juices is considered essential for both flavor and appearance, and any factors tending to cause instability in these attributes are considered undesirable. Stability can be attained by maintaining the product at a sufficiently low temperature. No significant changes were noted in cloud or flavor during 11 months' storage at 0°F. or below. At 5°F., change in cloud retention was slow but definite. At progressively higher temperatures, changes in cloud and flavor took place at progressively higher rates.

Probably the effects of storage at temperatures above 0°F. are cumulative and irreversible, and the effects of time and temperature must be considered whether it be in warehouses, during transportation, or in retail and consumer channels.

Safe Storage Conditions for Citrus-Pulp-and-Molasses Feed Defined

In the processing of citrus fruit for juice or frozen concentrate, two important byproducts are produced. These are citrus pulp, a dried material used as a feed, and citrus molasses, obtained from the liquid residue. The molasses is a thick liquid and very difficult for the dairyman or cattleman to handle. By drying the molasses with the pulp, a feed of uniform quality, easy to handle, is obtained.

However, certain questions arose in connection with this procedure. First, the industry needed to know how the addition of citrus molasses would affect the keeping of the citrus pulp. Also, because the addition of molasses increased the soluble-solids content of the dried feed, a method of analysis was needed so that a product of uniform composition and feeding value could be produced.

In an effort to answer these questions, the Citrus Products Station in Winter Haven, Fla., has made a thorough study of citrus pulp enriched with citrus molasses

The addition of citrus molasses did not introduce any serious storage problem. Lots with added molasses stored practically as well as those without. The studies revealed also that no difficulties would be expected in storing these feeds at relative humidities of 70 percent or less, but at 80 percent relative humidity the samples became moist enough to mold and spoil.

The Citrus Products Station also developed a method for estimating the soluble-solids content of feeds made from citrus pulp. Dried citrus feeds have always contained considerable soluble solids, the amount varying somewhat with the process used, condition of the fruit, and other factors. The addition of citrus molasses increases the soluble-solids content, and, with proper factory control by using the method developed, a more uniform product can be produced. The method is simple and rapid, yet is accurate enough for routine control work. Feed mills are using it to advantage in their operations.

VII. PINE GUM, TURPENTINE AND ROSIN

Continuous Distillation of Cleaned Pine Gum

A continuous steam still having a stripping column 32 inches in diameter and a maximum capacity of 5 tons of cleaned pine gum per hour was developed and thoroughly tested by the Naval Stores Research Division for commercial use. The operation of a 12-inch continuous steam still was reported in 1948. The additional work with the 32-inch still was done to achieve the present capacity, which is equal to that of the larger batch stills in the industry and is believed to be the maximum desired by any processor of pine gum. The rosin produced in typical continuous runs with the new still had a satisfactory melting point and was low in volatile oil, and the turpentine had a low acid number. To realize the maximum benefits of continuous distillation with this equipment, commercial plants would operate 24 hours each day. Continuous distillation would result in a 50-percent saving in steam consumption, reduction in labor and other operating costs, high-quality rosin and turpentine, and less variation in product quality. Completion of this new still ends nearly 10 years of process-development research.

Complete engineering data for the still can be furnished, on request, by the Naval Stores Research Division in Olustee, Fla.

Improved Practices in Gum Cleaning

To help processors who use the Department's public-service patents on gum cleaning meet the demand for high-quality products, the Naval Stores Research Division this year supplied them with details on improved practices of gum filtration and washing and the handling of chips.

The filtration of crude pine gum by the gum naval stores industry is a phase of plant operation in which savings in labor, time, and material costs can be of great value. To obtain these improvements the Naval Stores Research Division, in cooperation with a filter manufacturer, modified a commercial self-cleaning filter to adapt it to pine-gum processing. The new-type filter requires only the part-time services of one man for operation, uses more economical filter media, and can handle the low-quality gum that gives trouble in the filters generally used by the industry. The capacity of the filter, comparable to that of the conventional types, is such that only one cleaning per day is normally required. The first commercial installation of the new self-cleaning filter has been made.

The attention of processors was called to the necessity for the thorough washing of gum during cleaning to produce high-quality rosin. Although a single washing in the Olustee process produces high-grade, brilliant rosin, such rosin may still contain traces of contaminating materials. A thoroughly clean rosin can be made by recirculation of the filtered, washed gum a second time through the wash tank.

A third simple step in cleaning operations is recommended to processors as a means of upgrading their pale rosin when there is a price differential in favor of the paler grades. It comprises separating the chip cleanings from the main charge and distilling them with a lower grade of gum. This slight but important

change in the regular procedure will insure getting the best grade of rosin that the charge can yield. If the chip cleanings were distilled separately they would yield rosin that is several grades lower than the grade of rosin yielded by the gum from which the chips were separated.

Peroxides from Turpentine

The production of peroxides from turpentine and their use as replacements for the peroxide catalysts generally used in making synthetic rubber have been reported previously. This development has defense significance because the usual catalysts are based on benzene, which is in short supply. On the other hand, turpentine is abundant. Out of the 271,880 barrels of gum turpentine produced last season, only a small part went into industrial uses. Replacement of the benzene derivatives by terpene peroxides in the manufacture of about 700,000 tons of synthetic rubber would consume about 50,000 barrels of turpentine. This quantity of turpentine would substantially increase the proportion going into industrial uses.

In laboratory-scale tests, the Akron laboratories of the Office of Rubber Reserve last year demonstrated the superiority of the terpene peroxides, such as pinane hydroperoxide and menthane hydroperoxide, over cumene hydroperoxide, which was obtained from benzene. These laboratories recently confirmed the superiority of pinane hydroperoxide on a pilot-plant scale. They prepared and tested approximately 3,000 pounds of "cold" rubber (synthetic rubber made at relatively low temperature), using pinane hydroperoxide produced by the Naval Stores Research Division. The reaction rates and maximum conversion obtained with pinane hydroperoxide were considerably higher than those realized with the same quantity of cumene hydroperoxide at each of the levels investigated. The properties of the rubber made with terpene hydroperoxide as the catalyst were equivalent to those of similar stocks made with cumene hydroperoxide. Commercial production and use of the terpene peroxide, which appears feasible, would have the advantage, under current conditions, of conserving benzene supplies.